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QUALITY CONTROL OF NIOBIUM SHEETS FOR CAVITIES – COST AND CAPABILITIES OF EXISTING TOOLS

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Abstract:

Radio-frequency (RF) resonators (or cavities) are commonly used in particle accelerators to accelerate beams of charged particles to high energies. Fermilab is presently in the process of building up the capabilities required for the production of Niobium-cavities. A major requirement for state of the art cavities is that the Niobium is of high purity and free of inclusions or cracks. Inclusions of the size of 100 µm are detrimental to the performance of these devices. The following discusses possible tools for surface analysis and quality control of the Niobium sheets delivered by the Niobium manufacturers as the raw material for our cavity production. Past experience has shown that eddy current scanning is a possible method for the quality control of Niobium-sheets. Therefore the cost and specification of eddy current analysis tools will be discussed first. Alternative techniques for quality control will be discussed as well as, in a very superficial way, other more sophisticated tools used in the research on cavity surface properties.

1 INTRODUCTION

To achieve the highest possible gradients in Niobium cavities it is required that the Niobium is of exceptional purity and void of foreign inclusions or mechanical defects. The purity requirement for state of the art Niobium for superconducting cavities is that for example the most common impurity, tantalum, should not exceed concentrations of 500 ppm. High purity Niobium is fabricated using several stages of electron beam melting, achieving a RRR~300. This method is very effective in removing low melting point inclusions. In the case of Tantalum (which has a 500 degrees higher melting point than Niobium) this procedure, especially when performed in a non optimal way, can be insufficient. The latter requirement is related to quenches as a result of eddy current heating in normal-conducting inclusions or defects. A simple model can predict the critical scale of normal conducting defects on a Niobium surface exposed to strong RF fields. The temperature in a small normal conducting spot of radius, r, as a result of heating power, Q, is:

$$T \approx \frac{Q}{2\mathbf{p}kr} + T_b \qquad (K)$$

The RF heating is approximately given with:

$$Q = \frac{1}{2} R_s H^2 \mathbf{p} r_0^2 \quad (W)$$
 (2)

Combining these two equations, setting $T=T_c$, $H=H_{sh}$ and $r=r_0$ yields an expression for the maximum allowable spot size r_0 :

$$r_0 \approx 4(T_c - T_b) \frac{k(T_c)}{R_s H_c^2} \quad (m)$$
 (3)

With T_c =9.2 K, H_c =200/ μ_0 A/m, T_b ~2 K as for a typical Niobium cavity and R_s ~10 m Ω , k~100W/m/K for a Cu-like normal conducting spot, r_0 becomes 10 μ m.

The inclusions' order of magnitude yielded by this rough estimate, that is $\sim 10\text{-}100~\mu\text{m}$, sets the main specification for the required surface analysis tool resolution.

Typically the Niobium for cavities is delivered in the form of ~ 3 mm thick, rolled sheets, that are degreased and cleaned by chemical etching and annealed for 1-2 hrs at 700-800°C in a vacuum oven at $\sim 10^{-6}$ mbar to achieve full re-crystallization and a uniform grain size of $\sim 50 \, \mu m$.

To detect possible inclusions and mechanical damage it is required to check the raw material before proceeding to the production of the cavities. Voids, cracks and scratches deeper than 15 µm are not tolerable in the Niobium sheet and should be detected as well. It should as well be taken into account, that removing 100-200 µm of material from the surface occurs during cavity preparation. Inner defects that locate close to the surface will be uncovered. This means that quality control should be done both at the surface and inside of the Niobium disc bulk (to ~0.3 mm depth). Furthermore the quality control

method should be non-destructive and fast (scanning time of one sheet less than one hour). Niobium sheets are typically of rectangular shape with dimensions 265·265 mm² and 2.8 mm thick.

2 EDDY CURRENT SCANNING

Currently eddy current scanning is considered the most suitable method for the quality control of Niobium-sheets. In its simplest form eddy current scanning consists of measuring the impedance (and phase) change of a coil (typically a solenoid with a ferrite core) driven with an AC current of a defined frequency as it screens a conducting surface. The sample surface acts back on the exciter coil through the mutual inductance and induces a voltage in the exciter coil, thereby producing an apparent change in the coil reactance. The phase change contains information about the depth of the eddy current source in the sample. The impedance change of the exciter coil is measured e.g. with a Maxwell-Wien bridge. The principle of eddy current testing is sketched in Figure 1. The perturbation of the "regular" eddy current pattern in and on the specimen induced by defects produces a very small signal over the back-ground. Often this signal is much smaller than the (unavoidable) fluctuations of the distance between the probe and the sample. It is therefore crucial for eddy current testing to control the probe distance, optimize the sensing coil(s) configuration(s), the exciter current, signal amplifications as well as the electronics to obtain a resolution in the sub-mm range. In industrial applications the required resolution remains often in the mm-range (and the emphasis is more on penetration depth).

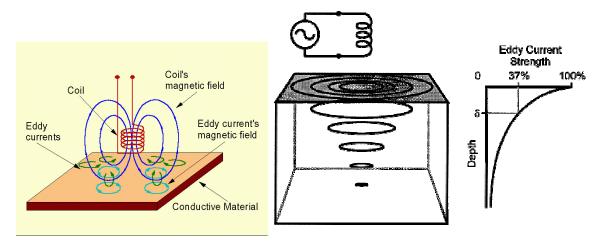


Figure 1: Principle of eddy-current testing (left), illustration of penetration depth (right).

Over the course of several years DESY has developed an eddy current scanner (see Figure 4) in collaboration with the german BAM institute in conjunction with the fabrication of cavities for TESLA. This eddy current scanning system uses a two-coil system (~3.5 mm diameter) in the absolute scheme (the differential scheme being optional) at two frequencies, 100 kHz and 1.5 MHz. The penetration depth in Niobium at 100 kHz is ~0.5 mm (see Figure 3). To reduce the effect of distance variations between the probe and

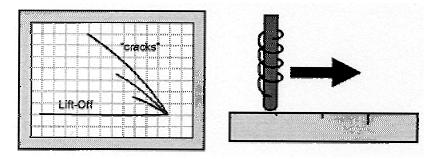


Figure 2: Signals obtained by eddy-current scanner passing over cracks of different depth (signal amplitude vs phase).

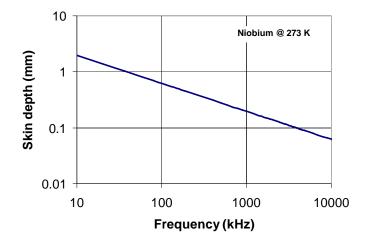


Figure 3: Penetration depth in Niobium at room temperature as a function of frequency.

the specimen, two systems have been used: 1) an air pressure pillow system (2 bar) and 2) an electromagnetic distance control system (Figure 6), the latter being in use in the most advanced prototype. In addition the Niobium sheet is pressed to the stretching table by pumping out the air under the sheet. In the course of the eddy current scanner R&D, sheets with drilled holes (down to 0.1 mm diameter) and artificial Tantalum defects (covered and un-covered) were prepared and tested. The method was optimized by extensive use of other experimental analysis techniques such as SEM, AES, EDX and NAA (described in more detail in part 4). The calibration using the above mentioned techniques revealed that the DESY system is capable of resolving 300 μ m Ta and 100 μ m Fe inclusions. JLAB has recently purchased an eddy current system of the DESY type (Figure 5) from FER-PA/Germany (a spin-off from the DESY-BAM development project) for the SNS cavity production. An example of a typical result obtained at DESY on "state of the art" Niobium discs for FNAL cavities is shown in Figure 7.

According to personal communications from experts at DESY (A. Brinkmann) and JLAB (G. Myneni) the quality of the raw material provided by selected vendors (such as Wah Chang, Heraeus, Denkai,...) today is such, that it is seldom that defects can be detected with eddy-current scanners of the above described type. JLAb has recently screened over 500 Niobium discs without detecting one single flaw. The experts at DESY pointed out that the device has been very useful in the past, when the Niobium production and



Figure 4: X-y (left) and rotating (right) eddy current scanners developed for DESY.



Figure 5: Turn-key eddy current scanner as purchased by JLAB from FER-PA GmbH/Germany.

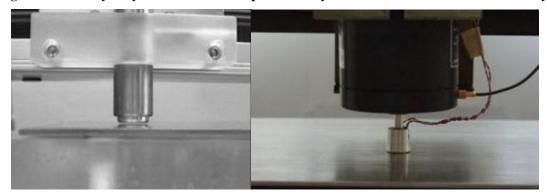


Figure 6: Pneumatically (left) and electro-magnetically (right) levitated probe-heads for the DESY eddy current scanner.

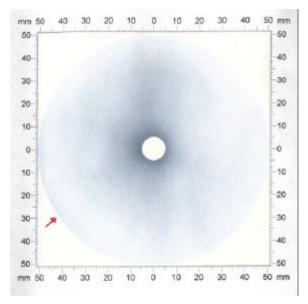


Figure 7: Example of eddy current scan performed on a Niobium disc for Fermilab by DESY.

purification procedures were not as optimized as today and that it might well be very useful for the screening of material from "new" vendors. It has to be noted as well that this should not be used as an argument for eliminating this quality control step. Rather, this means that, in view of higher gradients and better quality cavities, it might be recommended to look for techniques or devices yielding an even better spatial resolution. A specification of an eddy current scanner for Niobium sheets was issued to industry (see appendix 2). The specification was modeled after the scanner that was recently delivered by FER-PA to JLAB. Preliminary cost-estimates of US-firms can be found in Table 1. Although, the cost-estimates are very rough, they seem to indicate that the cost of the FER-PA product is reasonable and does not contain the cost of development.

Company	System	Cost (\$)	Remark
FER-PA, Germany	Turn-key	140 k	As purchased recently by Jefferson Lab
Sierra Matrix, CA	?	160k-210k	Rough estimate, includes development
MAC, NY	ROTOMAC9	64 k	Does not include vacuum sheet holder, depth resolution insufficient, probe distance control system not adequate, spatial resolution probably not adequate
IBG, Farmington Hills, MI	Turn-key	75-125 k	OK/NOK function only, no data archival involved
SDI, Newbury Park, CA		60 k	"SDI has built systems similar to the one you describe for ultrasonic and eddy current application"
R/D-Tech , Quebec, CAN		?k	in preparation – limits of resolution being investigated

Table 1: Cost estimates for turn-key eddy current scanner for Niobium sheets.

3 SQUID SCANNING

SQUIDs are being very successfully developed for NDE applications, especially for detecting defects in aerospace and civil engineering structures. In addition to their outstanding sensitivity to magnetic fields (to find magnetic inclusions or detect variations in magnetic properties), SQUIDs are useful because they can operate at much lower frequencies (<1 kHz) and can therefore detect defects far below the surface of a metallic structure. This is especially attractive, because it may allow post-production screening of the cavities from the outside.

SQUID microscopy was first commercially implemented in 1992. The extraordinary magnetic field sensitivity of SQUIDs is in the range $\sim 10^{-11}$ (for high T_c SQUIDs) to $\sim 10^{-13}$ T/Hz^{0.5} (for low T_c SQUIDs). This figure of merit relates a field resolution to the measurement time (frequency), i.e. at 10^{-11} T/Hz^{0.5} a measurement time of 0.1 msec (10 kHz) results in a resolution of 1 nT. The $f^{1/2}$ dependency in this expression is indicative of white noise, which typically depends on $1/f^2$.

A rough estimate of the signal strength can be made on the basis of (4), assuming the defect to be either a magnetic dipole with magnetic susceptibility χ or an eddy current loop induced by a given $d\phi/dt$.

$$B^{defect} \sim \frac{\mathbf{m}_0}{4\mathbf{p}} \frac{2m}{r^3} \quad (T) \quad m = MV^{defect} \approx \mathbf{c} \frac{B^{exciter}}{\mathbf{m}_0} V^{defect} \quad or \quad m \approx \frac{d\mathbf{f}}{dt} \frac{1}{R} A_{coil}$$
 (4)

The field produced by a spherical $(V\sim 4\pi(10^{-5}\text{m})^3)$ ferromagnetic defect $(\chi\sim 100)$ magnetized in the earth's field $(B^{exc}\sim 0.05~\text{mT})$, at the location of the SQUID (distance r~1mm) produces a signal of ~10 nT. A 10µm paramagnetic defect $(\chi\sim 10^{-4})$ would produce ~0.01 pT. Therefore the ferromagnetic can be differentiated from the Niobium background. Tantalum on the other hand, which has a similar χ as Niobium is much harder to detect. The relative magnetic susceptibility determines the background. Additional DC fields of the order of μ T in typical SQUID microscopes can increase the resolution.

AC exciter fields can be used to apply narrow band filtering to reduce the impact of external noise, and, applied at different frequencies allow to determine the depth of a defect. The surface eddy current response field to a ~T/sec exciter field at the μT level is (assuming A_{coil} given by 100 μ m pick-up loop and a resistivity of ~15·10⁻⁸ Ω m such as for Niobium at ambient temperature) at a coil distance of r=1 mm is ~0.2 pT. The most important issue in this regard is how much the signal differs from the back-ground, i.e. the remnant magnetism in Niobium and/or the cultural noise as well as the earth's field in non-shielded cases. SQUIDs, however measure relative fields, i.e. constant (or white noise) back-ground fields can be usually eliminated.

The spatial resolution of a SQUID system is determined by the diameter of the pick-up loop, with a few tens of microns being the cutting edge. On the other hand the smaller pick-up surfaces reduces the sensitivity. In fact, the product of coil-size and sensitivity is ~constant, such that these two parameters are to be traded off against each other. Another "golden rule" for optimum resolution is that the distance between the sample and the pick-up coil (or SQUID) should be comparable (~ 3 times) to the coil-diameter. SQUID

systems with cold samples (sample within the same cryogenic container as SQUID) have achieved µm level resolution.

There are many different SOUID based NDE techniques, such as simple magnetic field measurements or using eddy currents such as in traditional EDC, with the difference that the SQUID sensor is much more sensitive to the eddy current magnetic fields. Therefore the exciter fields may be applied parallel to the surface to reduce its effect on the SQUID. The SQUID sensor has to be cooled to cryogenic temperatures (and the sample should stay warm in our case), introducing a gap between sensor and sample which in turn reduces the strength of the eddy current signal. In systems using low T_c SQUIDs the minimum distance between sensor and sample of the order of 7 mm. High T_c SQUID system require less sophisticated cryogenic systems and can be brought closer to the sample (~0.5 mm). Since the sensor usually works in an unshielded environment and might even be exposed to the fields of the exciter coil, techniques have to be applied to reduce the "background" signal. These techniques are to measures 2nd and higher order derivatives of the magnetic field ("gradiometer arrangement"), to use multiple sensor coils and subtraction schemes, using electronic filtering in the form of flux locked loops (which is a standard technique for SQUID read-out anyway), using software based correction, ..etc. High T_c SQUID have to battle higher rates of thermal noise. Simple magnetometer configurations have been made to work as well in un-shielded environments. The SQUID output essentially consists of the strength of the magnetic field at the SQUID location (or in the pick-up coil that is coupled to the SQUID). An important limitation for high T_c SQUID systems is that the exciter fields, in case they are used, cannot exceed some ~mT. Unlike the traditional EDS method the SQUID technique does not naturally produce information about the depth of the signal. AC exciter coils operated at various frequencies are usually employed to generate the depth information. Another issue in the case of SQUID scanners is scanning speed. To obtain read-out rates of the order of 10⁴ points/sec (~0.5 m/s scanning speed) the time-averaging of the signal will be poor and thus the sensitivity reduced. According to the above relation a 1 MHz readout rate results in a reduced sensitivity limit of 10 nT, several orders of magnitude larger than the actual signals.

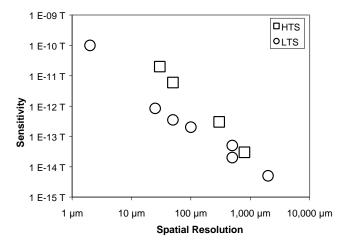


Figure 8: Sensitivity vs. spatial resolution of a number of Tristan SQUID microsopes.

A schematic of such a SQUID microscope is shown in Figure 9. Neocera Inc. sells a SQUID system in the US for \$ 380 k, Tristan sells its LTS SS QM for \$ 500 k (Figure 10). NTT and Sieko are competitors in Japan. FITM (now Fino-AG) and JSQ are European competitors.

We have contacted Tristan (Bob Fagaly) and found the SMM-770 to be a suitable device (except for the lack of automatic turn-table operation). These devices, however are very expensive. The operational principle of the Tristan SMm-770 is shown in Figure 11. The sensitivity plot (Figure 8) was supplied by Tristan as well.

First experimental evidence at DESY has shown that Fe-inclusions smaller than 50 µm and deep within the Niobium-sheet where easily detected with a prototype SQUID scanner, whereas it remained un-noticed with standard ECS. These commercial devices need to be adapted to the particular task of scanning niobium discs. Measurement speed, distance control, spatial resolution and optimization for the type of defects expected are

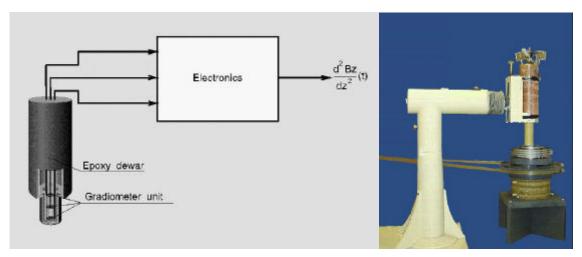


Figure 9: Schematic (left) and photograph (right) of SQUID gradiometer (courtesy of FinoAG/GR).



Figure 10: TRISTAN SQUID microscope SMM-1000, the world's first commercial magnetic microscope.

R&D issues. Therefore DESY has recently launched a ~\$ 1M R&D program, supported by the german ministry of science and involving FINO-AG and the university of Giessen, aiming at a fast and reliable SQUID scanner for TESLA (Figure 9). DESY plans on using the SQUID scanner in two modes: 1) to detect magnetic inclusions and 2) based on the thermo-electric effect a temperature gradient induced between bottom and top surface of the specimen causes thermal currents, with the associated magnetic fields being detected by the SQUID-based pick-up system. Other groups use the SQUID scanner in conjunction with a regular eddy-current scanning set-up in which eddy currents are excited in the specimen with a coil. Very common is the use of a "double-D" coil geometry, that allows to eliminate the excitation field from the SOUID reading. JLAB is close to launching a 3-4 year R&D program in collaboration with a low T_c SQUID vendor, a Niobium manufacturer, several universities (University of Virginia) as well as a private company that will commercialize the final product, aiming at the development of a SQUID based quality control system. The choice of modus operandi of this device has not yet been made. It is planned, however, to investigate several options, such as SQUID-arrays and gradiometer configurations The proposal for this R&D program, drafted by G. Myneni and P. Kneisel of JLAB is given in appendix 1.

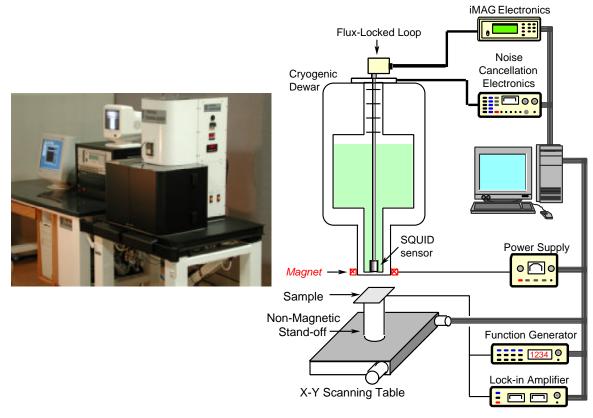


Figure 11: Photograph / schematic of Tristan SMM-771 magnetic microsope.

4 OTHER BULK AND SURFACE ANALYSIS TOOLS

Other, complementary methods that are useful for the identification of defects/inclusions and that have been used in the SCRF community are:

- SEM, scanning electron microscopy, most important tool, good but destructive, only surface, available at the Tech Division, related is the FESM (field emission scanning microscope);
- EDX, energy dispersive x-ray analysis: good, usually destructive and only surface, limited sensitivity (~0.5 % Ti);
- XRF, X-ray fluorescence, X-ray fluorescence is based on the principle of excitation of fluorescence with high intensity, wide wave-length range synchrotron radiation. In the synchrotron radiation fluorescence analysis (SYFRA) the excitation is done with a white beam and the fluorescence spectrum is analyzed with a semiconductor based detector. In the XAFS topography method the energy selection occurs in the primary beam and the absorption edge of definite elements is observed. The fluorescence method usually has a surface resolution of <100 microns and can be very sensitive (ppb-range). The method is limited to surface analysis only. The characteristic lines for Ta are: Ta-Kα1=57.532 keV, Ta-Kα2=56.277 keV, Ta-Kβ1=65.223 keV;
- X-ray radiography, Difference in x-ray absorption between defects and Niobium, can be destructive, complete penetration, "shadow picture" (good detection of elements with Z strongly different from Niobium), using film or liquid crystal detectors;
- AES, Auger electron spectroscopy, is the most common tool for quantitative surface analysis (resolution level: 0.1 % concentration, spatial resolution: μm). Evans provides commercial AES analysis (\$ 150/hour for "collaborations" with academia).
- NAA, neutron activation analysis (irradiation of sheet with neutrons, after formation of short lived isotopes measurement of emitted gamma radiation with Ge detector, the knowledge of the half-life of non-Niobium based isotopes can serve to identify these inclusions ¹⁸²Ta for example has a half life of 115 days, much longer than that of e.g. ⁹⁴Nb which is 6.2 min);
- ultrasonic reflection;
- neutron radiography;
- micro-hardness measurement is a good complementary tool to differentiate between benign defects such as scratch marks and real inclusions, due to the small penetration depth of the diamond pyramid (10 μ m) this test is also non-destructive.
- XPS, essentially a surface element analysis via work-function, i.e. INFN Genova, photo-electron spectrum analysis (energy, angle), standard X-ray source (MgKα, AlKα, keV energy range), nm depth resolution;
- X-ray laser interferometry, CEA, Saclay, exotic;
- PIXE, ion induced X-ray emission, Energy dispersive photon-counter, measurement of characteristic absorption lines of different elements, no depth resolution (few μm), semi-quantitative;
- ERDA, elastic recoil detection analysis,
- GDS, glow discharge spectroscopy, luminous emission of neutral particles from plasma created by glow discharge;

 TOF-SIMS, time-of-flight secondary ion mass spectroscopy, slow surface abrasion with ion beam combined with mass spectroscopy of released surface material, very high resolution (ppm range);

5 CONCLUSIONS

If Fermilab is seriously committed to building up a surface analysis capability in view of a production of superconducting cavities it would be recommended to acquire some form of cavity raw material quality control tools. Eddy current scanners, which are now common-place at cavity production sites (as well as Niobium manufacturers), have been developed and a turn-key type apparatus can now be purchased for ~\$ 150k. It is not advisable to invest into a development in-house. Although DESY is willing to lend support in the form of advice as well as free Niobscan software it is not certain if money can be saved by buying the minimum hardware from industry (probes and electronics) and designing/procuring the auxiliary hardware (vacuum sample clamping system, turntable with radial probe-arm and position monitoring, PC and cards and chassis) in-house. The cost of the turn-key EDS mentioned above has been cross-checked with offers from US-industry, indicating that it is reasonable and that no development cost is included.

DESY and JLAB have indicated the willingness to continue supporting us with eddycurrent scans of discs for the FNAL cavity production, as long as the number of samples to be scanned remains as small as it is today. They are as well open to the possibility that we perform scans on their facilities (being idle most of the time). These are probably the most straight-forward, short term solutions to the problem.

As indicated above, Niobium disc quality control will require a 10-100 µm spatial resolution (that is one order of magnitude better than standard ECS) in the future. Therefore DESY and JLAB have or are engaging in SQUID scanner technology R&D. Fermilabs money might be better invested into a SQUID development project, e.g. by joining the JLAB effort, with the perspective of having a better device in the end. Joining the JLAB proposal would probably strengthen it to the point where it can be launched. The terms of such a collaboration have not been discussed and certainly need to be defined. Best for us would be a in-house development of a complete system, e.g. by a PhD-student, taking advantage from the collaboration in the form of cheaper hardware (SQUIDs, Nb-samples,...) and the exchange of information. The cost of such a project has not been estimated yet, but it can be assumed to be less (not counting human resources) than the investment into a commercial SQUID microscope (which is not yet optimized for our needs), which would require ~\$ 500k.

Other methods of surface/ bulk analysis such as SEM, AES, XRF are necessary tools, but cannot be used on a routine basis. It is therefore preferable to occasionally outsource required measurements to private companies.

APPENDIX 1

JDRAJFT PROPOSAJL

Development of a Squid-based Scanning System for QC of RRR Niobium Sheets

G. Myneni, P. Kneisel

Objective:

Develop a system for scanning curved surfaces with the capability to detect defects in high purity Niobium of sizes much smaller than 100 micron.

2.1.1 Background

The ultimate performance limitation in a superconducting Niobium cavity is the transition from the superconducting to normal-conducting state, a "quench". It has been widely recognized and experimentally verified by temperature mapping [1] that quenches are caused by defects in the superconducting material. At the defect – either normal-conducting or weakly superconducting – more rf power is dissipated, which leads to an increase of the surface temperature in the surrounding area of the defect . Eventually a larger area heats up above the critical temperature of the material and a transition to the normal-conducting state takes place. Such defects can be foreign material inclusions, delaminations, cracks, surface scratches, welding beads, chemical residue...[2].

Thermal model calculations have shown [3,4,5] that the Niobium material can be thermally stabilized in the presence of defects by improvement of the thermal conductivity. The achievable field level is proportional to the square root of the thermal conductivity and inversely proportional to the square root of the defect size and defect resistivity

$$H_{\text{quench}} \sim \text{sqrt} \left[\kappa ? \right] / R_D x r_D$$

with $\kappa \mathbf{T}$) = thermal conductivity, R_D = defect resistivity and r_D = defect radius.

These model calculations indicate that defect sizes smaller than 50 micron are needed to achieve quench fields of ~ 100 mT, corresponding to accelerating gradients of ~ 25 MV/m in multi-cell cavities, if Niobium of RRR-values of > 250 is used [4] (the corresponding thermal conductivity is $\sim 60-65$ W/ K cm) [3]

A method of scanning the high purity Niobium sheets as supplied by the manufacturer has been developed over the last several years at DESY for the TTF cavities by using the eddy current method[6]. By applying this QC procedure and sorting out suspicious Niobium sheets, DESY and its cavity manufacturers have been widely successful in producing 9-cell cavities with gradients averaging $E_{acc} \sim 25 \, \text{MV/m}$ [7].

SNS has adopted this screening philosophy for its production cavity manufacturing and a system as shown in figure 1 and figure 2 has been procured and is being implemented at JLab.

The eddy current method has one intrinsic set back: the resolution of the method is limited to defects of a size not smaller than 100 micron; the limitation in resolution is resulting from the finite dimensions of the excitation and detector coils in the eddy current sensor.

Therefore, if one is interested in detecting defects of much smaller sizes, the elimination of which are necessary in achieving higher gradients (for a "defect-free" cavity the obtainable fields are \sim 180 mT at 1300 MHz corresponding to $E_{\rm acc} \sim$ 42 MV/m [8]) , a method with higher resolution must be employed. Such higher resolutions can be achieved with squid-based detection systems and initial developments have been started at DESY [9,10]. In first experiments an iron particle of < 50 microns was detected, which was not detectable with the eddy current scanning method.

Squid based system have been developed at various laboratories for material studies [e.g.11]; the resolution required for detection and elimination of defects < 100 micron in Niobium sheets can definitely be provided by such a system. It will have at least two advantages over the presently applied eddy current scanning method:

- a. detection of defects much smaller than 100 micron (depending on the obtainable resolution, it might be used for a large area pre-screening tool to look at grain boundary segregation, which then can be investigated with surface analytical instrumentation)
- b. scanning of curved surfaces, especially formed half cells (with the presently available systems, the Niobium is scanned before forming of half cells and any embedded defects during the forming and machining steps are "ignored")

The development of such a system poses enough challenges that it could be offered as PHD thesis theme.

Rough cost estimate

3 Squid System for initial evaluation	\$ 20.000
Scanning System (mechanics)	\$ 20.000
Computer, electronics	\$ 20.000
Data acquisition (software development)	\$ 10.000
Labor (4 man years)	

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APPENDIX 2

EDDY CURRENT SCANNER FOR NIOBIUM SHEETS - SPECIFICATION

Radio-frequency (RF) resonators (or cavities) are commonly used in particle accelerators to accelerate beams of charged particles to high energies. In the past, RF-cavities were made of copper. Today superconducting Niobium is becoming the favorite material for RF cavities. Fermilab is presently in the process of building up the capabilities required for the production of Niobium-cavities.

A major requirement for state of the art cavities is that the Niobium is of high purity and free of inclusions or cracks. Inclusions of the size of 100 µm are detrimental to the performance of these devices. For example the most common impurity, tantalum, should not exceed concentrations of 500 ppm. Today's Niobium production uses several stages of electron beam melting to achieve high purity. Typically the Niobium is delivered in the form of several mm thick, rolled sheets, that are degreased and cleaned by chemical etching and annealed at high temperature in a vacuum to achieve full re-crystallization and uniform grain size. The following contains a specification of an eddy-current test system that we plan to use to detect foreign inclusions such as tantalum or iron exceeding ~100 µm in the Niobium sheets delivered to us by the Niobium manufacturers as the raw material for our cavity production. Furthermore the eddy current scanner should allow detection of voids, cracks, scratches deeper than 15 µm. It should as well be noted, that 100-200 µm of surface material is removed during cavity preparation. Inner defects that are located close to the surface will be uncovered. This means that quality control should be done both at the surface and inside of the Niobium disc bulk (to ~0.5 mm depth). Furthermore the quality control method should be non-destructive and fast (scanning time of one sheet less than one half hour). Niobium sheets are typically of rectangular shape with dimensions 265-265 mm² and 2.8 mm thick. Past experience has shown that eddy current scanning is the most suitable method for the quality control of Niobium-sheets. The sheets will subsequently be cut to a round shape before being processed into cavities. Figure 12 shows a photograph of a superconducting cavity for a european particle accelerator.



Figure 12: Superconducting radio-frequency cavity for particle accelerator.

Table 2 contains the main parameters of the eddy current scanner. This list of parameters is commented in detail in the following.

Minimum defect size resolution	100 μm
Measurement mode	absolute AND differential
Frequency channels	100 kHz and 1.5 MHz
Depth resolution	0.5 mm
Step of position change of probe	200 μm
Probe lift-off	Non-touching (pneumatic, electromagnetic,)
Scanning speed	10000 points/second
Scanning mode	Rotating, continuous at ~0.5m/s
PC Control	yes

Table 2: Specifications for eddy-current scanner.

The samples consist of either rectangular or round, degreased and chemically polished sheets of thickness varying between 1 mm and 5 mm. The stretching table to which the samples will be attached should accommodate the thickness range mentioned above. Although the diameter of the Niobium disc is typically 25 cm, the sample-holder should be compatible with specimen up to 50 cm diameter. Niobium is a soft metal. Therefore the eddy current probe should not touch the specimen during screening. As indicated in Table 2 pneumatic or electromagnetic techniques are good candidates for levitating the probe from the disc surface because both methods allow control and regulation of the distance between probe and sample. The distance between the sample surface and the probe should be as small as possible. In addition it is preferable to avoid clamping the samples with mechanical devices to prevent damage to the discs and eliminate regions that are not accessible by the probe. Best suited would be a vacuum system, where by pumping out the air from beneath the sheet the disc can be fixed to the sample-holder, with the additional benefit of smoothening possible surface wrinkles on the specimen. As indicated in Table 2 the measurement should be performed while the sample is rotating at constant speed of ~0.5 m/s, with a speed chosen such that a measurement is made approximately every 200 µm along the track. The radial movement of the probe should be smooth as well ("spiral pattern") such that the distance between the spiral tracks is not more than 200 µm. Measuring with the sample in constant motion reduces the effect of inertia during acceleration of the probe-head that would be present in the case of the probe moving from one measurement point to the next. In addition this measure speeds up the procedure. The measurement time per sheet should be less than 0.5 hrs. It is as well preferable to use only one probe-head instead of multiple probe heads to prevent variations in the data sets due to differences in the probe settings. Furthermore, the effect of probe movement on the signal (moving cables...) should be reduced by amplifying the probe signal at the head itself. The system should be able to detect inclusions, cracks, ..etc larger than ~100 µm. The probe and measurement electronics can be of any design as long as they fulfill this requirement. A two frequency setting is required to allow screening of the bulk as well as the surface. The operating frequencies should be close to (but do not have to be exactly at) the values indicated in Table 2. The machine should be operated through a software allowing tuning of the main machine parameters, such as the probe settings (exciter current, amplification), the turn-table settings (speed), the data acquisition system settings, the pumps and power supplies. The program should display the measured data in real time as well as plot the signal amplitude according to a gray-scheme onto a plane representing the sample surface. The coordinate representation should be both in terms of x/y and r/φ. The software should allow zooming in onto a part of this plane (in both the measurement data and amplitude representation). Furthermore it should allow positioning of the probe to any point of the sample and the screening of a selected fraction of the sample surface.